SUPER ADSORBENT-BASED REMEDIATION AND ON-SITE FLOW INJECTION ANALYSIS DETERMINATION OF ARSENIC IONS IN GOLD MINING SITE

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SPECIAL DEDICATIONS TO:

My beloved parents, wife and children Yousif, Tiba, Mohammad, and Toqa, supportive siblings and to all my friends. May ALLAH always bless us now and forever.

My respected project supervisor

Prof. Dr. Abdull Rahim bin Mohd Yusoff

My dedicated co-supervisor

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Thanks you for always helping and supporting me

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ABSTRACT

Arsenic is a mineral which is abundant in gold mining sites. It is a toxic substance which needs to be removed. Thus, this research investigated the problem of extremely high arsenic (As) concentration in effluent (water and slurry) from Selinsing gold mine site, Pahang, Malaysia and developed an effective operational remedial method using an adsorbent material (Ecomel). In this research, a new method for measuring As(III) and As(V), which exist in the contaminated soil and liquid effluents was developed using flow injection analysis (FIA) system, while the total As was analyzed using atomic fluorescence spectrometry (AFS) for the remediating samples. By using the FIA method, the As speciation detection limit of (S/N = 3) for both As(III) and As(V) were found to be 5 µg/L with standard deviation 2.2 (n = 20). It also showed a wide dynamic range coefficient of detection (R^2) of 0.999 for As(III) and 0.9989 for As(V). This portable analytical method was successfully applied for the determination of As speciation in the effluent collected from Selinsing gold mine site without further treatment. For the characterizations of slurry and adsorbent materials (Ecomel), X-ray diffraction (XRD), X-ray fluorescence (XRF), energy-dispersive X-ray spectroscopy (EDX), Field Emission Scanning Electron Microscopy (FESEM), Focus Ion Beam–Scanning Electron Microscope (FIB-SEM), Inductively Coupled Plasma Optical Emission Spectrophotometer (ICPOES), Elemental Analyzer (EA) and Brunauer–Emmett–Teller (BET) were used. In the case of liquid effluent from the tailing dam, the pH and cyanide concentrations were measured at 11.5 and 204 mg/L, respectively. However, the batch experiments clearly proved that As leaching from the slurry (contact time: 24 h, stirring speed: 200 rpm, and S:L ratio 1:5) was extremely higher at pH 11.5 compared to pH 2.5 and were measured to be 8,720 and 1,010 µg/L, respectively. The major contributors to the exceedingly elevated levels of As concentration in liquid effluents were attributed to alkaline pH, high cyanide and silicate concentration, as well as high oxidation environment. The batch experiments on Ecomel revealed that maximum adsorption capacity determined at initial pH 2.5 from the Langmuir-Freundlich isotherm model was found to be 704.7 and 122.7 mg/g for As(III) and As(V), respectively. These results indicated that Ecomel has high adsorbent efficiency, cost-effective and is suitable for in-situ and ex-situ remediation of highly concentrated As(III) and As(V) toxicants in aqueous solutions. For As standard solution with initial pH 2.5 and concentration of As at 1 mg/L, it was observed that 0.3125 g/L of Ecomel with 2 h contact time can adequately remove 97.0% of As(V) and 98.1% of As(III), respectively. As a conclusion, results from AFS showed that the treatment of As using Ecomel at initial pH 2.5 was the most proficient for remediation of liquid effluent and slurry.

ABSTRAK

Arsenik adalah mineral yang terkandung dengan banyak dalam lombong emas. Ia merupakan bahan toksik yang perlu disingkirkan. Justeru itu, kajian ini mengkaji tentang masalah arsenik yang tinggi di dalam kandungan efluen (air dan lumpur) daripada tapak lombong emas Selinsing, Pahang, Malaysia dan membangunkan satu kaedah pemulihan berkesan menggunakan bahan penjerap (Ecomel). Dalam kajian ini satu kaedah baru bagi pengukuran As(III) dan As(V) yang terkandung di dalam tanah yang tercemar dan cecair efluen juga telah dibangunkan menggunakan sistem analisis suntikan aliran (FIA), manakala jumlah As bagi sampel pemulihan telah dianalisis dengan menggunakan spektrometri pendarfluor atom (AFS). Dengan menggunakan kaedah FIA ini, had pengesanan (S/N = 3) penspesiesan As bagi kedua-dua As(III) dan As(V) masing –masing ialah 5 μg/L dan 2.2% (n = 20). Kaedah ini menunjukkan julat dinamik yang luas dengan pekali korelasi (R^2) 0.999 untuk As(III) dan 0.9989 untuk As(V). Analisis mudah alih ini telah berjaya digunakan untuk penspesiesan As di dalam efluen yang diambil dari lombong emas Selinsing tanpa memerlukan rawatan lanjut. Untuk pencirian buburan dan bahan penjerap (Ecomel), pembelauan sinar-x (XRD), pendarfluor sinar-x (XRF), tenaga serakan sinar-x spektroskopi (EDX), mikroskopi medan pemancaran pengimbasan elektron (FESEM), mikroskop fokus ion alur-pengimbasan elektron (FIB-SEM), spektrofotometer plasma gandingan aruhan pemancaran optik (ICPOES), penganalisa unsur (EA) dan Brunauer-Emmett-Teller (BET) telah digunakan. Dalam kes efluen cecair dari empangan tailing, ukuran pH dan kepekatan sianida masing-masing adalah 11.5 dan 204 mg/L. Walau bagaimanapun, eksperimen kelompok jelas membuktikan bahawa ketelarutan lesap As dari buburan (masa sentuhan: 24 h, kelajuan kacau: 200 rpm, dan nisbah S:L 1:5) adalah amat tinggi pada pH 11.5 berbanding pada pH 2.5, masing-masing dengan ukuran 8,720 dan 1,010 μg/L. Penyumbang utama kepada tahap kepekatan As yang sangat tinggi dalam efluen cecair adalah disebabkan oleh pH beralkali, kepekatan sianida dan silikat yang tinggi, dan di tambah dengan keadaan pengoksidaan sekeliling yang tinggi. Eksperimen kelompok menggunakan Ecomel menunjukkan bahawa kapasiti penjerapan maksimum pada pH awal 2.5 dari model isoterma Langmuir-Freundlich masing-masing adalah 704.7 dan 122.7 mg/g untuk As(III) dan As(V). Dapatan ini menunjukkan bahawa Ecomel mempunyai kecekapan penjerapan yang tinggi, kos efektif dan sangat sesuai digunakan untuk pemulihan *in-situ* dan *ex-situ* bagi larutan yang mengandungi bahan toksik As(III) dan As(V) dalam kepekatan yang tinggi. Bagi larutan standard yang mengandungi As pada pH awal 2.5 dengan kepekatan 1 mg/L, didapati bahawa Ecomel dengan kepekatan 0.3125 g/L dan dengan masa sentuhan 2 h, dapat menyingkirkan 97.0% As(V) dan 98.1% As(III). Sebagai kesimpulan, dapatan daripada AFS menunjukkan rawatan As menggunakan Ecomel pada pH awal 2.5 adalah yang paling berkesan bagi pemulihan cecair efluen dan buburan.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

AAS	-	Atomic absorption spectroscopy
ABS	-	Absorbance
AdSV	-	Adsorptive stripping voltammetry
AES	-	Atomic emission spectroscopy
AFS	-	Atomic fluorescence spectrometry
AFU	-	Arbitrary fluorescence unit
Al	-	Aluminum
AMD	-	Acid mine drainage
ARE	-	Average relative error
As	-	Arsenic
AS	-	Automated sampler
As(III)	-	Arsenite
As(T)	-	Total arsenic
As(V)	-	Arsenate
As_2S_3	-	Orpiment
AsS	-	Realgar
Au	-	Gold
B	-	Boron
BET	-	Brunauer–Emmett–Teller (BET)
BOFS	-	Basic oxygen furnace slag
BPC	-	Back pressure coil

C	-	Carbon
Ca	-	Calcium
CC	-	Cooling coil
Cd	-	Cadmium
CE	-	Capillary electrophoresis
ClO_2	-	Chlorine dioxide
CN^-	-	Cyanide
Co	-	Cobalt
CO	-	Column oven
COPD	-	Chronic Obstructive Pulmonary Disease
Cr	-	Chromium
CR	-	Coloring reagent
CS-P-NZVI	-	Nanoscale zero-valent iron on pumice
Cu	-	Copper
DO	-	Dissolved oxygen
DOE	-	Department of Environment
DTA	-	Differential thermal analysis
EA	-	Elemental Analyzer
EC	-	Electrical conductivity
EDX	-	Energy-dispersive X-ray spectroscopy
Eh	-	Redox potential
e-MWCNT/Fe^{2+}	-	Iron(II) oxide coated ethylenediamine functionalized multiwall carbon nanotubes
e-MWCNT/Fe^{3+}	-	Iron(III) oxide coated ethylenediamine functionalized multiwall carbon nanotubes
ER/DETA/FO/FD	-	Cross-linked macroporous polymer impregnated with hydrous iron oxide:
ET-AAS	-	Electrothermal atomic absorption spectrometry
Fe	-	Iron

Fe^0	-	Zero Iron
Fe^{2+}	-	Ferrous ions
Fe^{3+}	-	Ferric ions
FeAsS	-	Arsenopyrite
FeS	-	Pyrite
FESEM	-	Field Emission Scanning Electron Microscopy
FIA	-	Flow injection analysis
FIB-SEM	-	Focus Ion Beam –Scanning Electron Microscope
GC-MS	-	Gas chromatography-mass spectrometry
GFAA	-	Graphite furnace atomic absorption
H_2O_2	-	Hydrogen peroxide
H_4SiO_4	-	Silicic acid
HCN	-	Hydrogen cyanide
HG-AAS	-	Hydride generation coupled with atomic absorption spectrometry
HG-AFS	-	Hydride generation coupled with atomic fluorescence spectrometry
HIO-alginate beads	-	Hydrous iron oxide-impregnated alginate beads
HPLC-AFS	-	High performance liquid chromatography with atomic fluorescence spectrometry
HPLC-ICP-MS	-	High performance liquid chromatography-inductively coupled plasma mass spectrometry
IARC	-	International Agency for Research on Cancer
ICP-AES	-	Inductively coupled plasma emission spectrophotometry
ICP-MS	-	Inductively coupled plasma mass spectroscopy
ICPOES	-	Inductively Coupled plasma optical Emission spectrophotometer
KIO_3	-	Potassium iodate
$KMnO_4$	-	Potassium permanganate

L/S	-	Liquid to solid ratio
LC-MS-MS	-	High performance liquid chromatography-tandem mass spectrometry
LDL	-	lower detection limit
LOI	-	Loss on ignition
Mn	-	Manganese
Mo	-	Molybdenum ion
N	-	Nitrogen
N**	-	Not measured
$Na_2S_2O_3$	-	Sodium thiosulphate
NAA	-	Electrochemical methods, neutron activation analysis
NaClO	-	Sodium hypochlorite
NH_2Cl	-	Monochloramine
NIIEM	-	Nano-iron ion Enrich materials
$-NO_3^-$	-	Without NO_3^-
NSD	-	Normalized standard deviation
NST	-	New straits time
NZVI	-	Nano-zero-valent iron
nZVI in nZVI/BC composite	-	Nano-zero-valent iron in biochar supported zero-valent iron nanocomposite
nZVI/BC	-	Biochar supported zero-valent iron nanocomposite
NZVI-AC	-	Nano zero-valent iron on activated carbon
OM	-	Organic matter
ORP	-	Oxidation-Reduction Potential
PO_4^{3-}	-	phosphate
P1 & P2	-	Double plunger pump
pb	-	Lead
pH_f	-	Final pH value
pH_i	-	Initial pH value

pH_{pzc}	-	Point of zero charge
PHTEs	-	Potential hazardous toxic elements
PP	-	polypropylene
PW	-	Pure water 0.6 mL/min
PZD	-	Particle size distribution
R^2	-	Coefficient of determination
RC	-	Reaction coil
Redox	-	Reduction/Oxidation
RMSE	-	Residual root mean square error
RSD	-	Relative standard deviation
S	-	Sulfur
S/N	-	Signal-to-noise ratio
SDS	-	Sodium dodecyl sulfate
SE	-	Standard error
SiO_3^{2-}	-	Silicate
SPC	-	Spectrophotometer
STD	-	Standard deviation
Temp.	-	Temperature
TG	-	Thermogravimetry
TGA	-	Thermo-gravimetry analysis
Unimpregnated HIOs	-	Unimpregnated hydrous iron oxide
UPW	-	Ultra-pure water
WHO	-	World health organization
WTRs	-	Water treatment residuals
WW	-	Wastewater
XRD	-	X-ray diffraction
XRF	-	X-ray fluorescence
Zn	-	Zinc
ZVI	-	Zero-valent iron

LIST OF SYMBOLS

C_0	-	Liquid phase concentrations of As(III)/As(V) at initial time zero
C_e	-	Equilibrium concentration in the solution (mg/L)
C_t	-	Liquid phase concentrations of As(III)/As(V) at time t
E%	-	Percentage removal of arsenic
K_1	-	Pseudo-first order sorption rate constant (h^{-1})
K_2	-	Pseudo-second order constant (g/mg h)
K_f	-	Freundlich constant (L/g)
K_{id}	-	Intra-particle diffusion rate constant (mg/g $h^{0.5}$)
K_{L1}	-	Langmuir constant (mg/g)
K_{L2}	-	Langmuir-Freundlich constant $(L/mg)^{1/n}$
n	-	Freundlich adsorption equilibrium constant (dimensionless)
q	-	Quantity of adsorbed As(III)/As(V) at equilibrium settings (mg/g)
q_e	-	Quantity of As(III)/As(V) adsorbed on the surface of Ecomel at equilibrium (mg/g)
q_m	-	Maximum adsorption capacity (mg/g)
q_t	-	Amount of As(III)/As(V) adsorbed by Ecomel, at time, t (mg/g)
R_{L1}	-	Dimensionless equilibrium constant for Langmuir model
R_{L2}	-	Dimensionless equilibrium constant for Langmuir-Freundlich models
t	-	Time (h)
$t_{1/2}$	-	Time for half equilibrium (min)
V	-	Volume of the As-contaminated solution (L)

W	-	Mass of Ecomel used for As(III)/As(V) adsorption (mg)
α	-	Parameters of the Elovich rate equation (mg/g)
β	-	Parameters of the Elovich rate equation (g/mg)
χ^2	-	Chi-square test
n_1		Langmuir-Freundlich constant (dimensionless)

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Background of the study

Gold mining sites are mostly associated with positive economic development of any country; however extraction of gold could also generate negative impacts on the ecosystem and human health. Mining and ore-processing activities generate huge quantities of tailing that could have severe and widespread impacts on ecosystem over a long period of time [1]. Gold mining is considered as a significant source of environmental pollution by trace elements especially high arsenic (As) concentration arising from ore excavation, transportation, mineral processing, and disposal of huge quantities of waste around the mining site [2,3]. Recently, ecosystem deterioration is regarded as an important problem, and both active and expected mining exploitations are legally compelled to minimize the contamination impact and to restore the exploited sites after their abandonment. The serious environmental problem and public-health concerns associated with gold mining activities is As, which is released from the oxidation of sulfide minerals such as pyrite (FeS_2) and arsenopyrite (FeAsS) [4].

Gold is often occurring as encapsulated in other minerals such as FeS_2, quartz, FeAsS, etc. The particles of gold are generally extracted via ore leaching using cyanide (CN^-) ion solution. Throughout the cyanidation process, the metal ore

reacts with CN^- ion solution under highly alkaline conditions (pH > 11) for duration of up to 36 h, thus, releasing high As concentration. The As-bearing tailings from sulfide ore mines are usually chemically reactive because of their content of Fe-sulfide minerals, like as FeS_2 and FeAsS. The weathering of Fe-sulfide minerals is a strong acidifying process, and regarded to be the major reason for acid mine drainage (AMD) [5,6]. Arsenic can't form stable complexes together with CN^- ion such as gold (Au), however, it is well-known that As are very soluble at the highly alkaline conditions observed for the cyanidation process. Thus, the water used in this process is highly contaminated with dissolved As and CN^- ion.

Consequently, the huge amount of water used in this process must be remediated to remove dissolved As prior to its discharge into tailings dam. The management of As-bearing tailings generated through gold extraction processes usually concentrates on the stability of secondary As-phases mainly because As-sulfides such as, realgar (AsS), orpiment (As_2S_3), FeAsS are assumed to be oxidized during gold extraction. However, As could be existing in two major redox states, namely; arsenite As(III) and arsenate As(V) in aqueous effluent, both having differences in solubility and mobility according to the oxidation-reduction potential (ORP) and pH conditions in the ecosystem [7]. The major secondary As minerals existing in tailing dam after cyanidation process are supposed to be As(V), due to the addition of high hydrogen peroxide concentration during the gold extraction process. At oxidizing conditions, As(V) generally forms complexes with metals such as Fe, Al and Ca [7,8]. However, the stability and formation of these As(V)-metal complexes is based on several important factors, such as, pH, Fe/As or Al/As ratio, redox potential conditions, availability of O_2 and CO_2 as well as the presence of anions, such as SO_4^{2-} ion, phosphorus (PO_4^{3-}) and silicate (SiO_3^{2-}) that compete with As(V) for sorption surfaces [9,10]. Fe and Al-As(V) are the dominating phases in acidic condition during gold mining and are less soluble than Ca-As(V), such as schwertmannite or ferrihydrite. Gold mine tailings slurries after gold extraction containing As-enriched sulfide ores are usually remediated by lime and $Fe_2(SO)_3$ to

enhance the formation of stable Fe-As-precipitates and increase the solution to prevent the formation of acid mine drainage. Furthermore, As-bearing tailings slurries are usually pre-remediated with H_2O_2 and O_2 in order to enhance the oxidation of As(III) complexes into less mobile As(V) species [6,11].

However, in recent years the world have witnessed the emergent cases of As contamination in water and aqueous effluent as a big global environmental problem, associated the rapid growth of modern industries and mining process [12]. In many cases, the As concentration in aqueous samples from gold mining site are determined within the range of 10-100 mg/L at acidic settings, especially in FeAsS-rich areas [4,13]. Whilst in soil, As was detected ranging from 2,000-390,970 mg/kg in acid mine drainages [14,15]. These highly elevated concentration of As should be effectively remediated using highly efficient adsorbent to reduce its environmental impact.

Due to the high toxicity of As, the world health organization (WHO) announced the maximum permissible limit of As in drinking water to be 10 μg/L [16]. And also U.S. Environmental Protection Agency (EPA) adopted a lower standard limit from 50 to 10 μg/L of As concentration in drinking water [17]. Therefore, there is great concern for As contaminations for human health, plants, soil, and water to prevent the ecosystem from deteriorations caused by the high toxicity of As presence.

These increasing trend of growth of interest in As remediation is in agreement with the result of the study performed in 2007 where an estimate of more than 137 million people in over 70 countries, including Argentina, Bangladesh, China, Hungary, India, Japan, Malaysia, Mexico, Romania, United States and Vietnam are exposed As toxicity via water sources [18–20]. Thus, the great harm to human health for As was affirmed and for this reason, As was categorized as class

one carcinogen by the International Agency for Research on Cancer (IARC) [21]. Additionally, more than 200,000 people suffered death due to As poisoning every year after being subject to a variety of cancerous growths such liver, lungs, bladder, kidney and prostate cancers [22,23]. Similarly, As has also been related to non-cancerous multi-systemic health problems such as renal failure, diabetes mellitus, dermatological diseases, respiratory disorder, cardiovascular disease, hypertension and so forth [18,24]. Unfortunately, As contamination removal is not easy as well, as it can easily pass through food chains and drinking water into the body. Moreover, As exhibits a strong tendency for accumulation in the human body and hence threatens prolong health risks [12].

Due to gold mining processes, high As concentrations are generated for both tailing and aqueous effluents. These highly elevated As must be remediated before discharge into the water bodies. The remediation of As-bearing tailings or As contaminated area is a considerable challenge to the scientific community, especially at mining sites that have dramatically impacted the water resources and soils in the environment [25]. In other hand, As removal from aqueous effluent can be a very costly process. The costs for As removal could exceed the benefited, needing very careful selection of the technology to be used. First of all, the selection must consider the water characteristics to be remediated [26]. To date, there are various technologies applied to remediate As from natural waters and industrial effluents including coagulation-precipitation, oxidation, coagulation, ion-exchange, membrane separation, adsorption, reverse osmosis and electro-kinetic methods. However, these technologies have several drawbacks such as high waste remediation and operating costs, high consumption of reagents as well as large amounts of toxic sludge formation. Among these processes, adsorption technique is considered as one of the most promising technologies for mitigating As in aqueous effluent scenario, due to its low cost, operational simplicity, high concentration efficiency, and environmental friendliness [27,28]. Consequently, it is vital to properly remediate this aqueous

effluent utilizing techniques that involve, for example, adsorption in a complementary way or as alternatives for conventional techniques.

Nowadays, As remediation from aqueous effluent has become a major active research focus. Numerous approaches and studies are applied to obtain abundant, porous structure and chemically-stable inexpensive adsorbent with high surface area [18]. These includes adsorbents such as iron oxide, iron oxyhydroxides, aluminum oxide/oxyhydroxides, activated carbon, activated alumina, rare earth oxides, fly ash, manganese green sand, iron-containing mesoporous carbon, natural material, both Fe_2O_3 and Fe_3O_4 nanomaterials, red mud, zeolite, zero valent iron nanoparticles, granulated ferric oxide, manganese oxides, transition metal sulfides, magnetic wheat straw, titanium dioxide, iron-oxide@carbon and cellulose@Fe_2O_3 composites, etc. The effectiveness of adsorption technique selection is mostly dependent on the adsorbent's removal efficiency and their economic value. Due to the tremendous adsorption potential, magnetic properties, and strong affinity of iron toward As, iron oxide compounds or embedded iron have high sorption affinity toward As species, therefore, iron based materials are commonly used in various methods for the remediation of As contaminated water and soil [17,20,28].

1.2 Problem statement

Nowadays, many instruments used for measuring As includes hydride generation coupled with atomic fluorescence spectrometry (HG-AFS), hydride generation coupled with atomic absorption spectrometry (HG-AAS), electrothermal absorption spectrometry (ET-AAS), inductively coupled plasma with atomic emission spectrometry/inductively coupled plasma with mass spectrometry (ICP-AES; ICP-MS), high performance liquid chromatography with atomic fluorescence

spectrometry (HPLC-AFS), high performance liquid chromatography with inductively coupled plasma-mass spectrometry (HPLC-ICP-MS) etc. [29–31]. These analytical techniques cannot be considered as portable instruments for on-site applications as they required complicated sample pre-treatment process, and skilled personnel [32]. As(III) dominates the gold mining site due to FeS_2 or FeAsS oxidation. As(III) can commonly get oxidized and transformed to As(V) during the sampling to laboratory. Therefore, the result of the test is often biased and does not express the exact As speciation of the samples [33]. Consequently, there is a need to develop a simple, sensitive and accurate method for on-site analysis of As speciation in environmental water sources.

In the gold mining site, great amount of water is used for gold extraction For example, in Selinsing gold mining site, about 6000 m^3/d of effluent are discharged into the tailing dam. This aqueous effluent contained extremely high concentration of As and CN^- ion as well as high alkaline pH and oxygen concentration. Consequently, the water resources near the gold mine are contaminated. Thus, exposing the inhabitants to severe health risks such as arsenicosis, cancers, diabetes, hearing loss, hematological disorders, keratosis, neurobehavioral disorders, and pulmonary fibrosis [30,34,35].

In our visit to Selinsing gold mine site, the site engineer informed us that they had problem in the management of the aqueous effluent (and not the sediment which according to him is stable). We were informed that the As concentration in first tailing pond is generally between 10-17 mg/L which was extremely high in comparison with the effluent standard for As in Malaysia (0.1 mg/L).

1.3 Research objective

The main objectives of this research are as follows:

a) To develop a new technical method for measuring As speciation.

b) To determine the As concentration in soil and aqueous effluent from Selinsing gold mining site (Malaysia), and identify the problems associated with the elevated As concentration in its discharge effluent.

c) To investigate the applicability of Ecomel (Iron ion enrich materials) for removal of As(III) and As(V) in standard aqueous solutions and treating the slurry from gold mine site using batch method.

1.4 Scope of research

For measuring As speciation, redox–FIA system was developed. The remediation of As in liquid effluents was carried out using Ecomel under acidic condition without the addition of oxidants. The experimental flow of the research is shown in Figure 1.1. Thus, this research thesis was divided into three phases as follows:

Phase I: Developing new technical method for As speciation measurement in water samples co-existing with phosphorus (PO_4^{3-}) and silicate (SiO_3^{2-}) using FIA.

The first phase focused on developing new method for measuring As speciation and includes:

a) Investigation of the simple As speciation analysis for As (III), As (V) and in PO_4^{3-} and SiO_3^{2-} contaminated environmental sample.

b) Optimization of the oxidant concentration that can complete oxidation of As(III).

c) Optimization of the reductant concentration that can complete reduction of As(V).

d) Investigating techniques to eliminate the effect of PO_4^{3-} and SiO_3^{2} as interference to As analysis by molybdenum blue.

e) Testing the accuracy of Flow Injection Analysis (FIA) for measuring As based on comparison with atomic fluorescence spectrometry (AFS).

f) The system performance of FIA for As speciation determination in real sample.

Phase II: Sampling field works at Selinsing gold mining site in Pahang Malaysia

The second phase focused on obtaining the environmental assessment for the mining site as the background of the study. Activities done during this phase includes;

a) Case study of Selinsing gold mining site problems in aqueous effluent and tailings.

b) All aqueous effluent and As-bearing tailings were collected from Selinsing gold mining site at 17-7-2015 and 3-1-2016.

c) Characterizations of soil from tailing dam using XRD, XRF, pH, pH_{pzc}, FESEM-EDX, nitrogen-adsorption analysis, ICPOES, AFS.

d) Onsite water quality measurements such as, pH, DO, ORP, EC, and temperature with ICPOES, AFS, and As speciation by FIA.

e) Studying the characterizations of soil such as pH, particle size distribution, classification of soil and Loss on ignition (LOI).

f) Studying the effect of pH and L/S on As leaching.

Phase III: Batch experimental set using Ecomel adsorbent, a new application for As remediation in aqueous solution and slurry waste.

This phase focused on the preparations, testing and characterizations of Ecomel before and after adsorption. The processes include:

a) Characterizations using XRD, FESEM-EDX, pH, pH_{pzc}, TGA-DTA, nitrogen-adsorption analysis.

b) Adsorbent kinetics and isotherms.

c) Study the mechanism of As removal.

d) Cost analysis study for Ecomel.

e) Studying the optimum dosage of Ecomel for remediation As-bearing

tailings.

f) Studying the optimum dosage of Ecomel for remediation As(III) and As(V) from water.

g) Studying the optimum dosage of Ecomel for remediation As from slurry.

h) Model suggestion for slurry remediation.

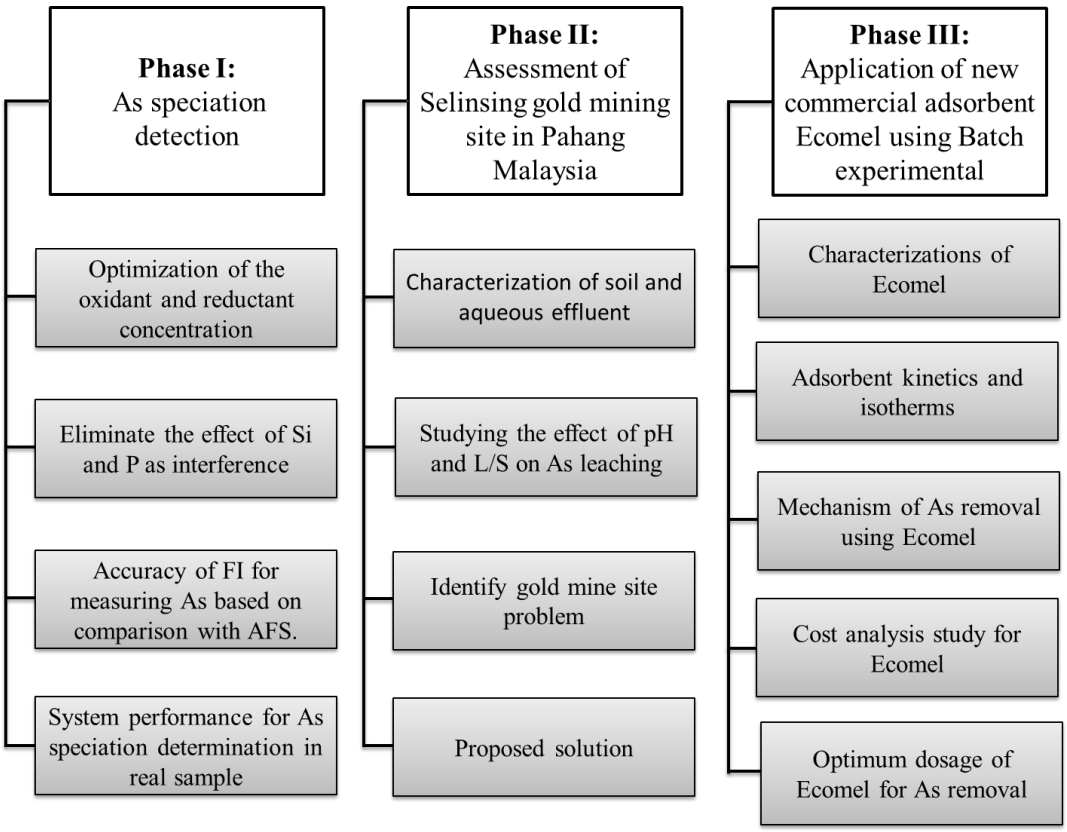

Figure 1.1 Experimental flow

1.5 Significance of research

The significance of this research is to resolve an actual problem in Malaysia and many other developing and developed countries resulting from high As concentration. This explores the viability of a proposed model for As remediation in aqueous effluent in gold mining site.

The present research focuses on Selinsing gold mining site, Pahang–Malaysia. Field studies were performed to identify the problem of high As leaching from tailing. Adsorption under acidic conditions in laboratory batch scale was used for the remediation of queues effluent. Generally, the present study provides a complete view on the cause of As leaching in aqueous solution and suggest the reliable system to remediate As and overcome any potential problems associated with As leaching.

Even though the aqueous effluents were remediated by adding hydrogen peroxide (H_2O_2) for the oxidation of As(III) to As(V) and as well integrating high concentration of iron impregnated sand to adsorb As(V), As concentration in treated effluents were still extremely high and does not meet Malaysia's As effluent discharge standards (0.1 mg/L). Thus, this study introduces a new application of super-adsorbent namely Ecomel with low-cost and high adsorption capacity to adsorb As especially As(III) without any further pre-remediation. Ecomel can remediate high As concentration in both aqueous effluent and slurry.

The success of this research will consider Malaysia as a contributing nation for sustainable mining policy and as well create higher reputation to the Malaysian's research activity and applied novelty of low-cost technology for on-site remediation in As-bearing tailings. This enables Malaysia to have within their domain, a recent

technology for remediating As from gold mining sediment and protect its water resources from As contaminations. Therefore, this study provides a promising alternative technique for the remediation of As-bearing tailings in view of its low-cost and long term application as a sustainable mitigation technology.

Similarly, a simple, selective and stable on-site Flow Injection Analysis (FIA) redox analytical method was developed to determine As speciation in water samples constituted with phosphate (P) and silicate (Si). The developed method senses the As(V), P and Si by reacting with ammonium molybdenum, which forms a molybdenum blue complex under acidic condition. This study was carried out to define an alternative method to remediate As contamination in aqueous effluent. As mobility in As-bearing tailings can be controlled by two important processes: (i) adsorption and desorption processes and (ii) solid-phase precipitation and dissolution.

1.6 Limitation of research

Briefly, the limitation of this research can be summarized by the following:

a) Difficulty to get information about the industrial process, concentration on the influent/effluent and adsorbent material that was used for As remediation in aqueous effluent in Selinsing gold mining site.

b) Limitation of sampling frequency due to stringent regulation from the management and the location of sampling site is far from UTM.

c) Difficulty to apply the research on-site for remediating both aqueous and slurry effluents.

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